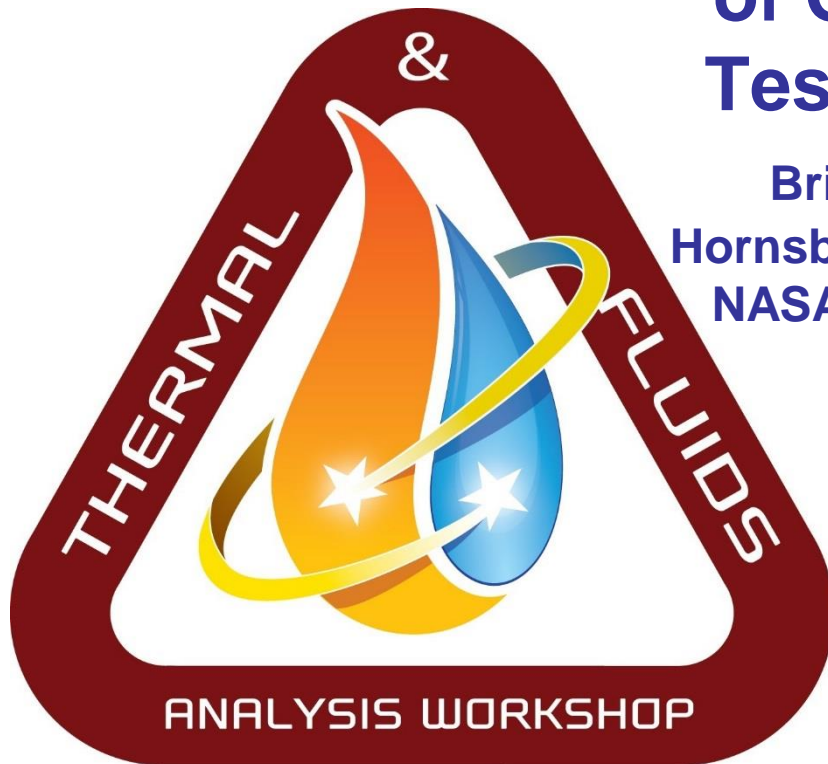




Development of NASA's Sample Cartridge Assembly: Summary of GEDS Design, Development Testing, and Thermal Analyses

Brian O'Connor, Deborah Hernandez, Linda Hornsby, Maria Brown, and Kathryn Horton-Mullins
NASA Marshall Space Flight Center and Jacobs Technology ESSSA Group

Presented By
Deborah Hernandez
Jacobs Technology ESSSA Group



TFAWS
MSFC • 2017

Thermal & Fluids Analysis Workshop
TFAWS 2017
August 21-25, 2017
NASA Marshall Space Flight Center
Huntsville, AL

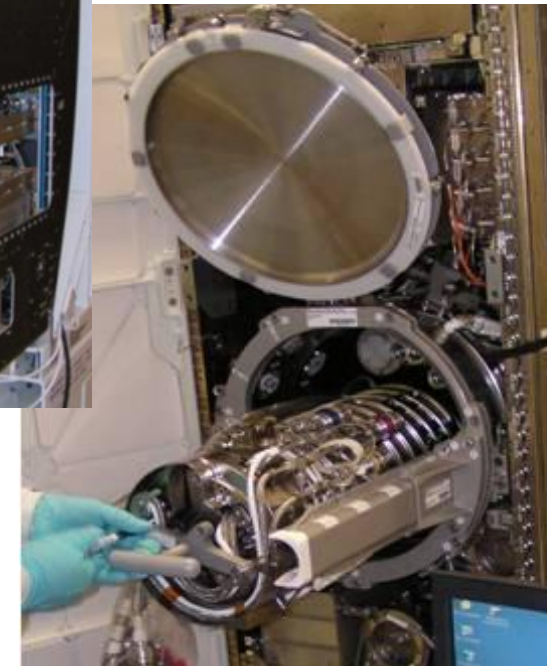


Outline



- Background of ISS Material Science Research Rack
- NASA SCA Design
- GEDS Experiment Ampoule Design
- Development Testing Summary
- Thermal Modeling and Analysis
- Summary
- Future Work
- References

- ISS MSRR1- Materials science research in low gravity
 - Destiny Laboratory, launched in 2009
- ESA's Material Science Lab (MSL) - Built by ESA
 - Process material samples at high temperatures
- Low Gradient Furnace (LGF) insert - Bridgman style furnace with multiple heater zones
 - Vacuum furnace
 - Two hot cavities separated by an adiabatic zone
 - Max operating temperature of 1400° C



- NASA SCA

- 26 mm diameter
- Argon gas pressurized volume
- Reusable after processing
- Qualification tested at 1294 °C

- Instrument Head

- 416 CRES
- Interface to MSL Intermediate Support Plate (ISP)
- Ultrahigh vacuum seal with bolted joints
 - Conflat flanges comprised of a copper gasket and knife-edge flange

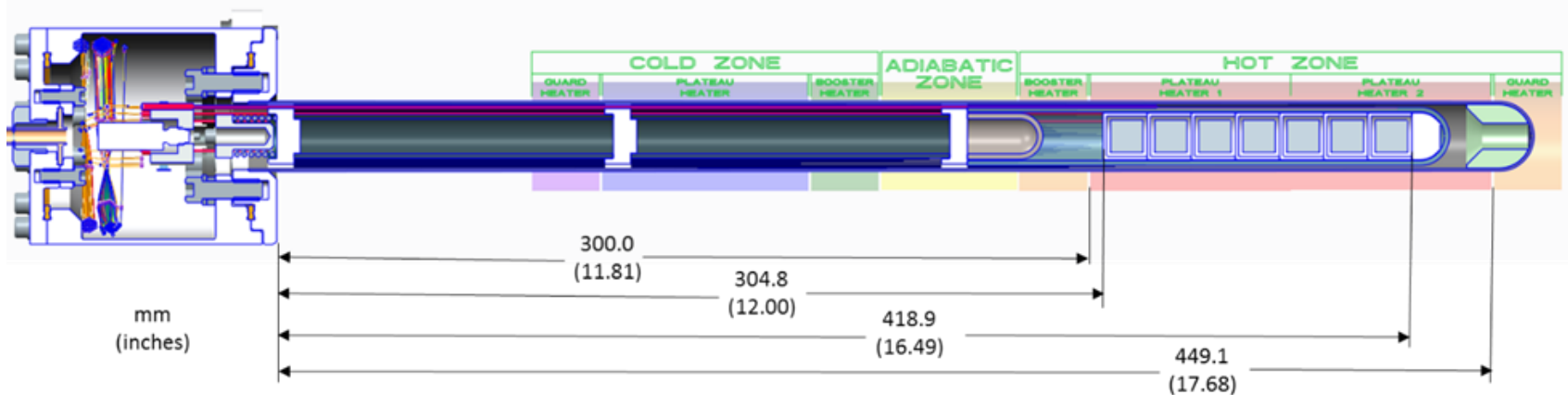


- Cartridge Tube

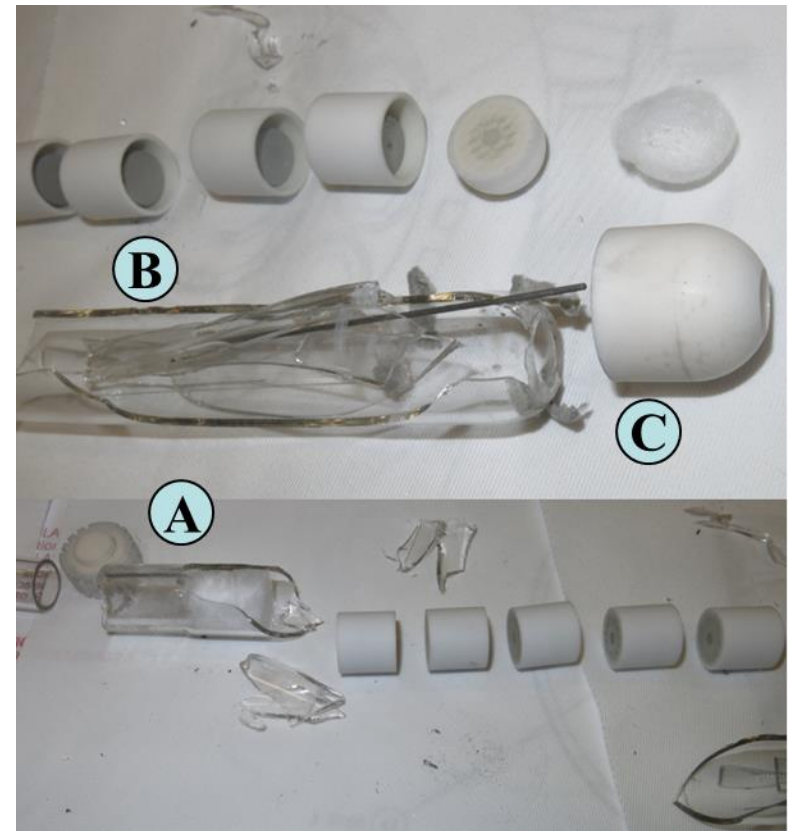
- Vacuum Plasma Sprayed
- Molybdenum-Rhenium with an Alumina liner and Zirconium Boride emissivity coating

GEDS Experiment Ampoule Design

- GEDS research (Dr. Randall German Principle Investigator)
 - Determine the underlying scientific principles on how to forecast density, size, shape, and properties for liquid phase sintering
- Seven samples in each GEDS SCA
 - Compacted mixture of W, Ni, Cu, and Mn powders
 - Samples located in LGF isothermal hot zone
 - Dwell duration above 1200 °C from 3 to 60 minutes
 - Sample stack isothermality of $\pm 5^\circ\text{C}$
- Repeatable processing profile for each of 7 SCAs
 - Controlled by LGF Sample Processing Program (SPP)
- Vacuum Ampoule
 - Cartridge filled with argon to prevent ampoule permeation
 - High purity alumina ceramic crucibles



- Development Test MUGS Results
 - The Tantalum Sheath of the Type N Thermocouples were disintegrated
 - Ampoule failure in three places by three modes
 - A. Ampoule chipped at alumina spacer interface
 - B. Ampoule body broken with marks at TC locations
 - C. Contact at the sphere ampoule end to alumina end plug



MUGS Ampoule Post Test



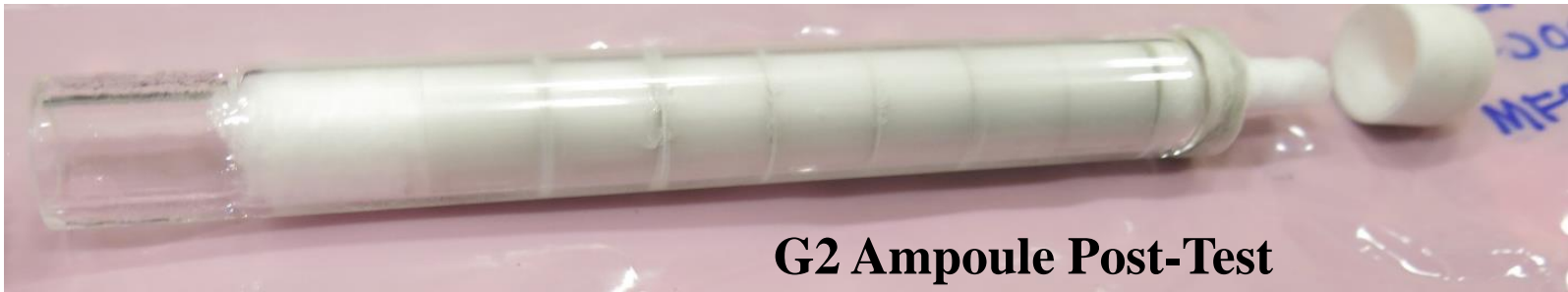
Development Testing Summary



	MUGS	MUGS V	MUGS VI	MUGS VII	MUGS VIII	G2
Test Date	July 2016	November 2016	December 2016	January 2017	March 2017	May 2017
Processing Duration at heater set points	22 minutes at 940°C/ 1210°C	78 minutes at 940°C/ 1210°C	78 minutes at 940°C/ 1210°C	78 minutes at 940°C/ 1210°C	18.5 minutes at 940°C/ 1210°C	95 minutes at 1130°C/ 1210°C
Predicted sample dwell time	9.3 minutes > 1200°C	66 minutes > 1200°C	66 minutes > 1200°C	66 minutes > 1200°C	3 minutes > 1200°C	60+ minutes > 1200°C
Design Revisions (design changes additive)	None	<ul style="list-style-type: none"> • Re-aligned ampoule to bottom of cartridge • Reduced TCs number • Added quartz wool to 0.375 inch thickness • Reduced spring force to 10# • Increase fill pressure 	<ul style="list-style-type: none"> • Upgrade ampoule quartz to 314C • No samples or crucibles • Reduced spring force to 8# 	<ul style="list-style-type: none"> • Added full ampoule back into SCA • Revised bakeout for sample and crucibles • Reduced spring force to 5.2# 	<ul style="list-style-type: none"> • 2 Type S platinum sheath TCs • Reduced spring force to 4.8# 	<ul style="list-style-type: none"> • Longer processing time • 4 Type S platinum sheath TCs • Increase cold zone setting from 940°C to 1130°C
Test Result	Ampoule failure in 3 modes	Ampoule failure in 1 mode	Ampoule intact	Ampoule intact, less sintering than expected	Ampoule intact	Ampoule intact

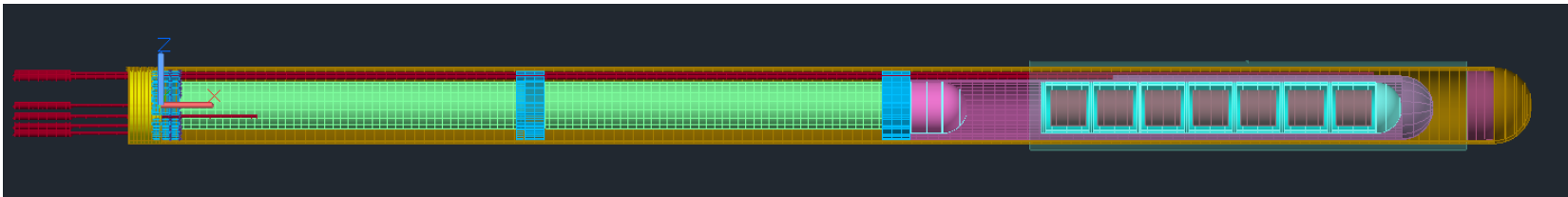
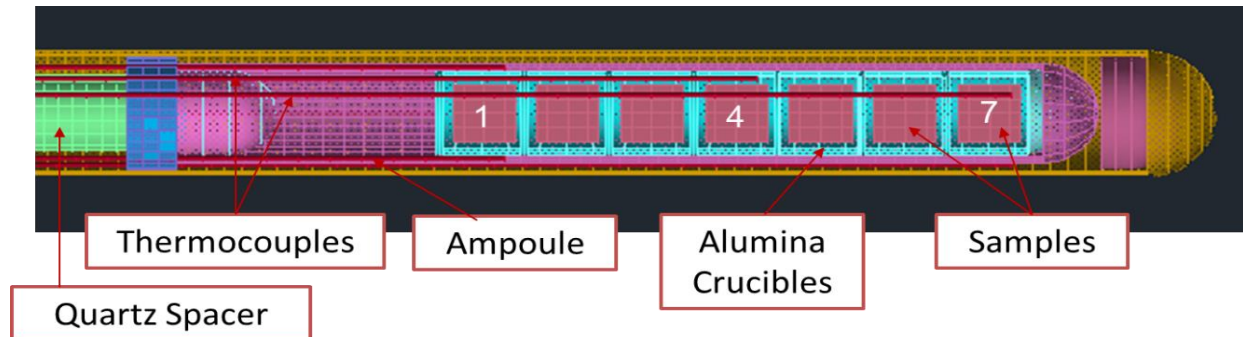
Design Change Summary

- SCA internal design
 - Reduced TCs number from 8 science TCs to 4 science TCs
 - Reduced spring force 10# to 4.8#
 - Added quartz wool to 0.375 inch thickness
- GEDs Ampoule
 - Re-aligned ampoule to bottom of cartridge
 - Upgrade ampoule quartz to 314C
 - Revised bakeout for sample and crucibles
- Processing Changes
 - Increased processing time from 78 minutes to 95 minutes
 - Increase cold zone setting from 940 °C to 1130 °C

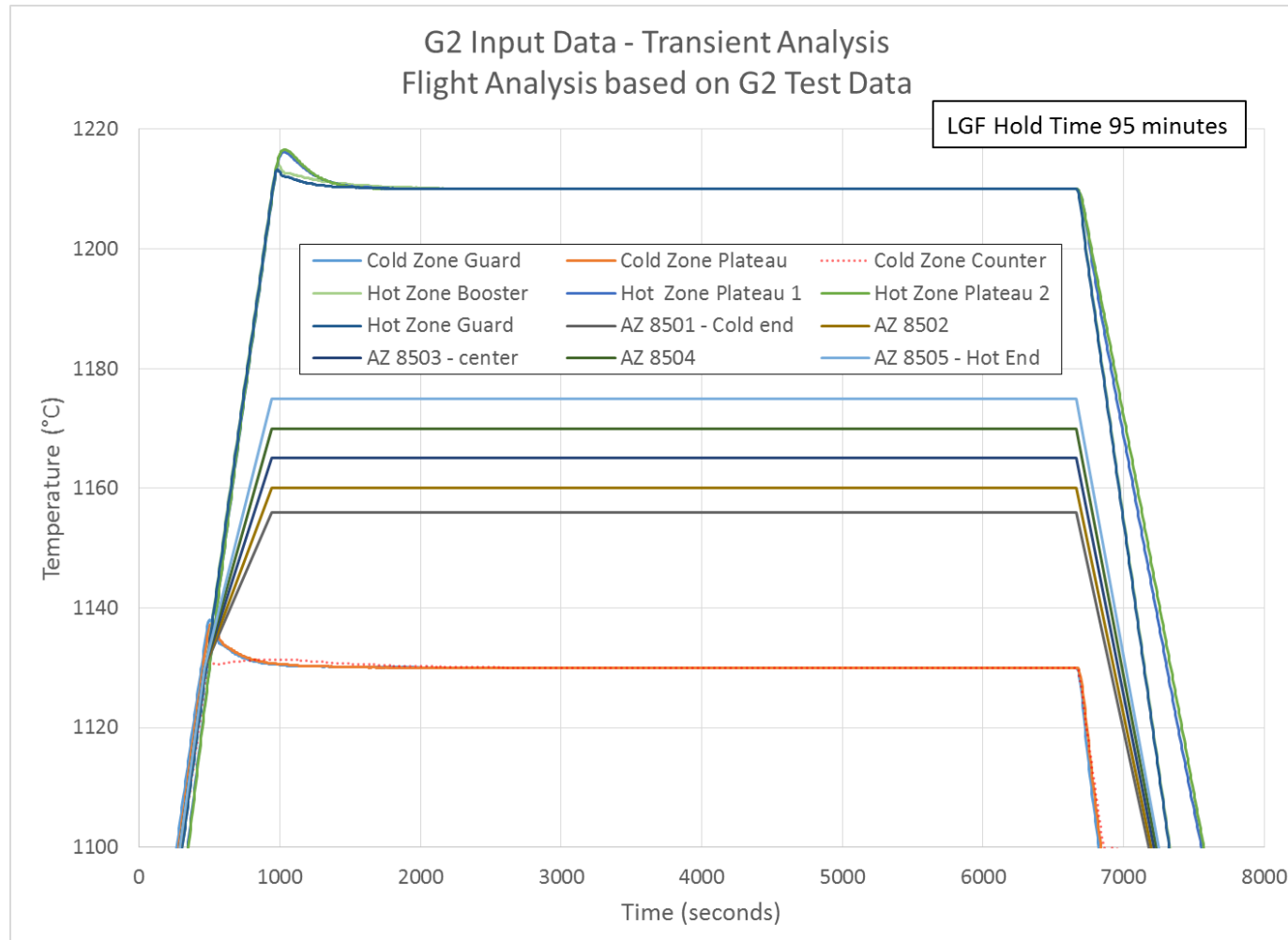


G2 Ampoule Post-Test

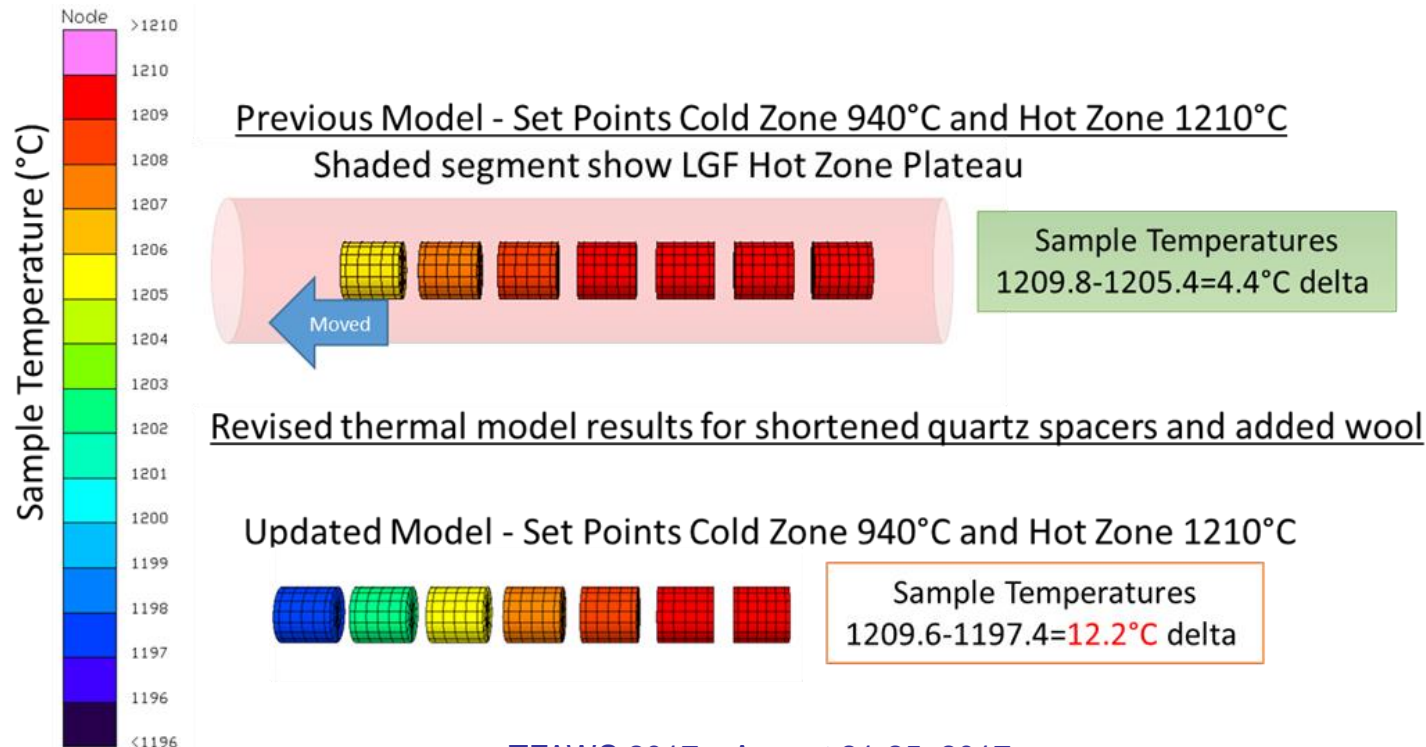
- Atypical transient analysis performed to simulate LGF response
 - Understanding the LGF transient response is critical to GEDS experiment success
 - LGF thermal model was not applicable for transient analysis
- Thermal model development
 - Represents only internal components of GEDS SCA cartridge
 - No simulation of sample sintering



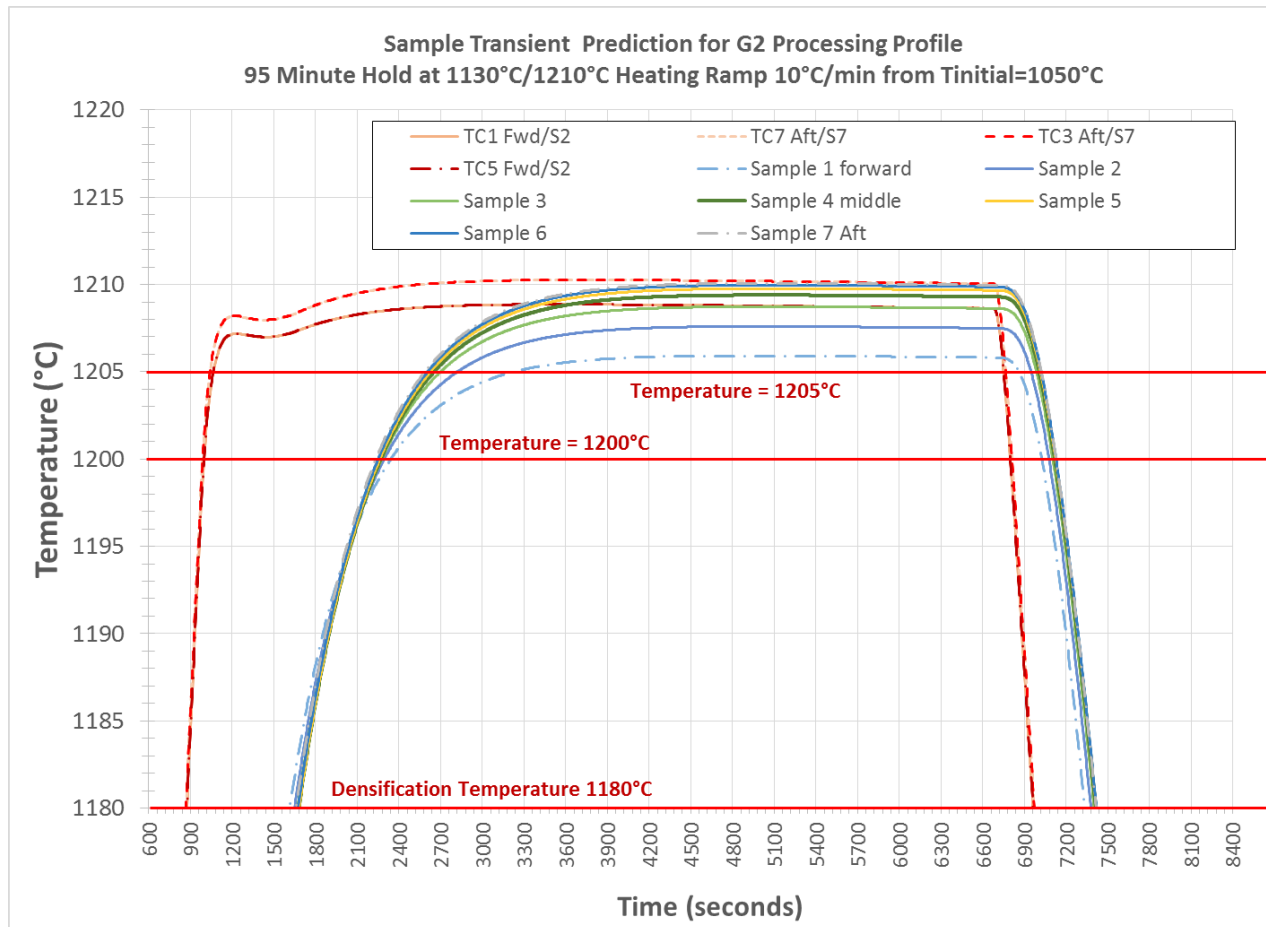
- Heated zone profile based on MUGS development test data to simulate LGF heating transient

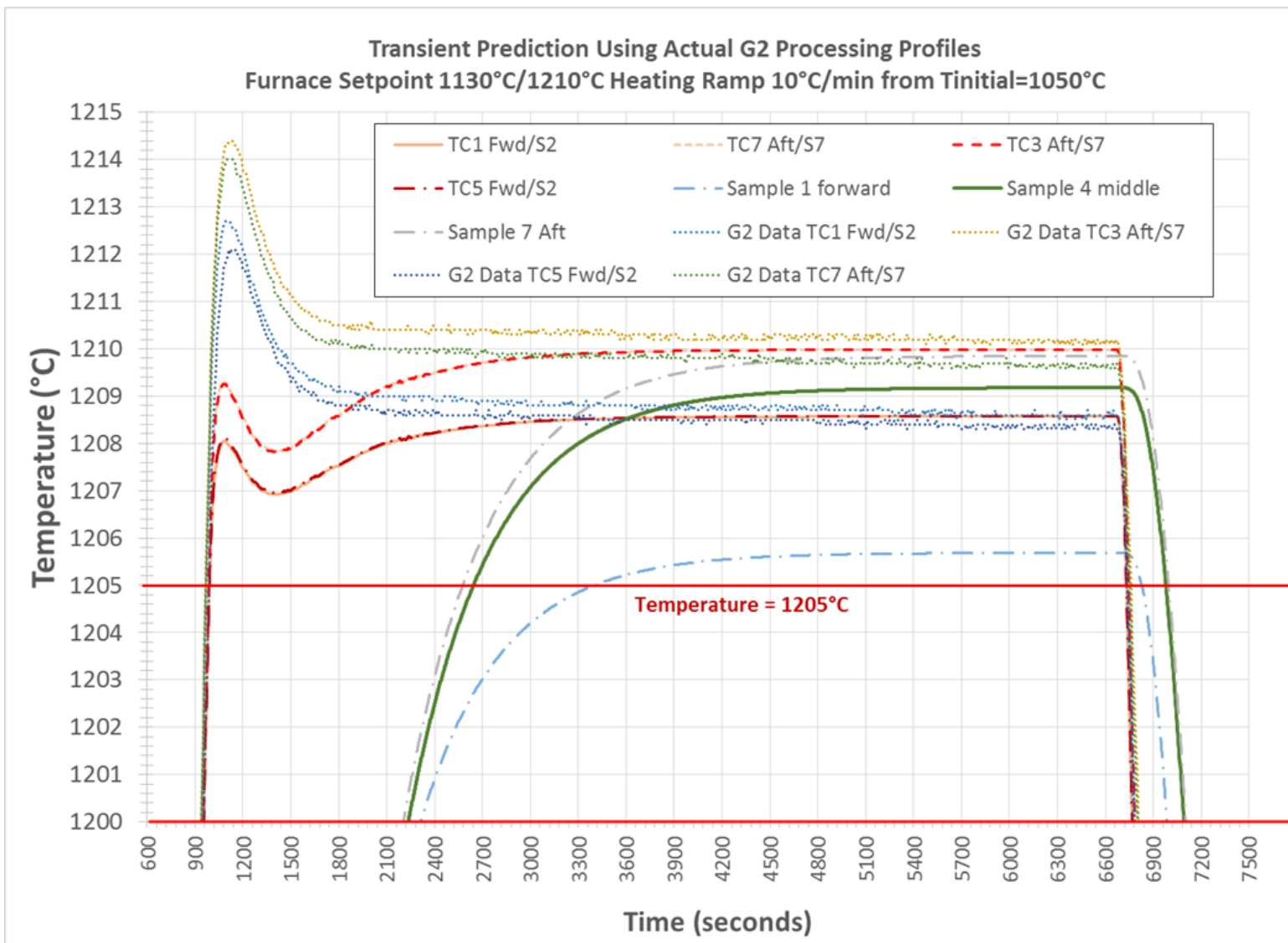


- Thermal model over predicted sample dwell time in the sintering range
 - PI MUGS sample evaluation showed long duration samples were not fully sintered
- MUGS thermal model based on initial GEDS design
 - Design changes shifted samples about 0.5 inches toward the adiabatic zone with MUGS test design changes integrated
 - Unable to increase hot zone setting above 1210°C due to design limitations

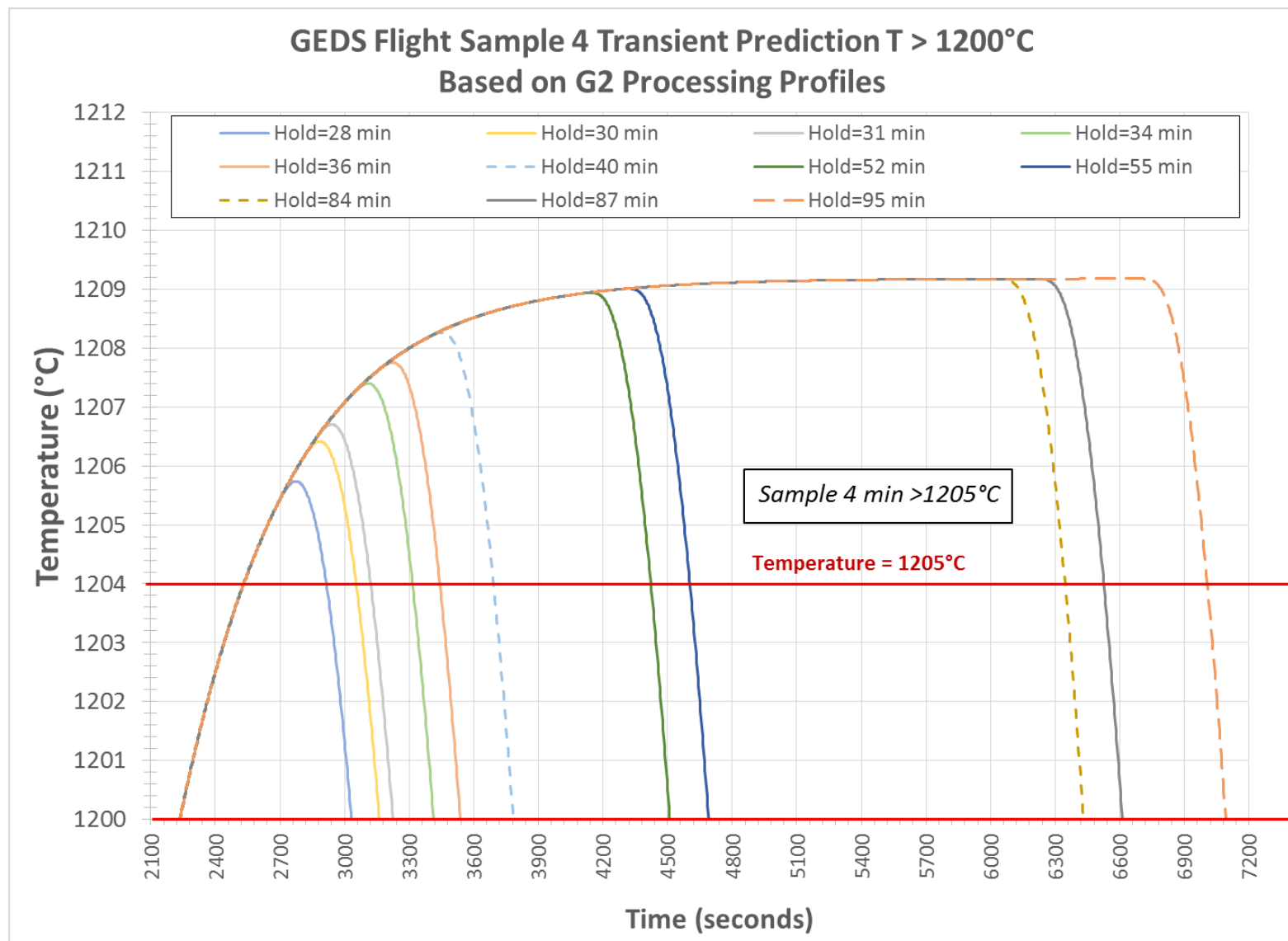


- Used updated transient model to revised processing profile prior to ground preflight test, G2
 - Model updated to Increase cold zone temperature to 1130°C to prevent end effects
 - Sample 1 dwell time = 60 minute calculated base on time > 1205°C





Predicted Sample Flight Processing



- GEDS design development challenges
 - GEDS Ampoule design developed through MUGS testing
 - Short duration transient sample processing
 - Unable to measure sample temperatures
- MUGS Development testing used to gather data
 - Actual LGF like furnace response
 - Provided samples for sintering evaluation
- Transient thermal model integral to successful GEDS science
 - Development testing provided furnace response
 - PI evaluation of sintering anchored model predictions of processing durations
 - Thermal transient model used to determine flight SCA processing profiles

- MSFC SCA features
 - 26 mm diameter
 - Argon gas pressurized volume
 - Cartridge is reusable after processing
 - Qualification tested at 1294 °C
- Future SCA experiments
 - Influence of Containment on the Growth of Silicon-Germanium (ISCAGE)
 - Cadmium Telluride (CdTe)
 - Both require directional solidification at a controlled gradients



References



- 1) <http://msrr.msfc.nasa.gov/>. [Online]
- 2) *Simulation of ESA's MSL Furnace Inserts and Sample-Cartridge Assemblies: Model Development and Correlation with Experimental Data.* Johannes Dagner, Marc Hainke, and Jochen Friedrich. Rome, Italy: 35th International Conference on Environmental Systems, 2005.
- 3) *Development of NASA's Sample Cartridge Assembly: Design, Thermal Analysis, and Testing.* B. O'Connor, et al. Bellevue Washington: International Conference on Environmental Systems, 2015.
- 4) *Characterization of Vacuum Plasma Spray Formed Molybdenum-Rhenium Alloys.* J. Scott O'Dell, et al. Orlando, Florida: International Conference on Tungsten, Refractory & Hard Metals VI, 2006.
- 5) *Multiscale Modeling and Experimentation on Liquid Phase Sintering in Gravity and Microgravity Environments, MSRR-1 SCA Science Requirements Document (SRD), MSRR1-DOC-0115,* Dr. Randall M. German, MSFC, Alabama, January 24, 2014.
- 6) T. Panczak, S. Ring, M. Welch, D. Johnson, B. Cullimore, D. Bell. *C & R Technologies (R) Thermal Desktop (R) User's Manual, A CAD Based System for Thermal Analysis and Design, Version 5.8.*